CONTROL OF MATERIALS

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SEDIMENTATION ANALYSIS OF DISPERSE MATERIALS

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A scanning photosedimentograph with a high accuracy of determination of the granulometric composition of materials is described, which has increased measurement reliability and durability due to the absence of a mechanical drive and the presence of eight measuring channels. Original mathematical software is developed, which allows for automatic determination of integral size distribution of particles.

In some technological processes (production of certain types of ceramics, grinding powders, etc.) the granulometric composition of finely milled materials is one of their main parameters. Moreover, the dispersion composition of materials to a large extent affects the operation of diverse types of technological equipment, for instance, aspiration equipment and pneumatic transport systems for friable material. Consequently, the need for estimating the granulometric composition of materials makes the development of methods and instruments for dispersion analysis topical.

There are various methods for granulometric analysis of finely milled materials, many of which are considered in detail in P. A. Kouzov's study [1]; moreover, the traditional methods of analysis are constantly being refined.

New instruments are represented by laser analyzers, whose principle is based on radiation diffraction by particles. However, despite several advantages (fast analysis, wide measurement interval from 0.1 to 1000 μm) these instruments have some disadvantages as well. The problem is that material together with the dispersion medium in the course of analysis is pumped via a cuvette. As a consequence of the abrasive effect, the surface of the cuvette is worn, becomes turbid, and fails. This is especially typical of such material as grinding corundum micropowder.

In this case it is preferable to use the classical photosedimentograph, in which the medium in the cuvette remains immobile during analysis and the rate of particle sedimentation is low. As a result, the service life of the cuvette in such device is substantially longer than in a laser meter. That is why some foreign companies along with laser analyzers continue manufacturing instruments of this class. It should be noted that the design of the photosedimentograph is being constantly upgraded as well, which contributes to maintaining its competitiveness. Unfortunately the domestic industry has not yet started mass production of measuring devices of this class.

The design of photosedimentograph SF-2 developed at the Ural State Technical University can be of certain interest. In contrast to its foreign analogs, it has some fundamental distinctive features that contribute to increased reliability of measurements and a longer service life.

The foreign sedimentographs (for instance, Analysette-20 produced by Frich) have a mechanical drive for scanning the cuvette along its height. The drive, like any other mechanism, lowers the reliability of the device in general and may lead to additional measurements errors. The main distinctions of the photosedimentograph SF-2 consist in the absence of a mechanical drive and the presence of eight measuring channels. The cuvette with a suspension inside is scanned by eight sensors located at different heights.

The device SF-2 (Fig. 1) consists of a stand *1*, into which the cuvette *2* is inserted. Eight radiators *3* and eight receivers *4* are mounted on the stand. The radiators are IR light diodes AL107A having monochromatic radiation in the IR range with a wavelength of 0.9 μm. The receivers are IR photodiodes KFDM. The use of the IR range makes it possible to completely eliminate the effect of light interference. Power to the radiators is supplied via a current-stabilized power unit (PU). A signal from the generating photodiodes arrives at the input of the eight-channel operational amplifier (OA) and, after amplification, at the input of the analog-digital transducer (ADT). The signal in the digital form is transmitted from the ADT via the interface RS 232C to the successive computer port COM1 or COM2. The set is equipped with special software including the following main blocks:

– a block for mathematical processing of measurement results with approximation of integral distribution using vari-

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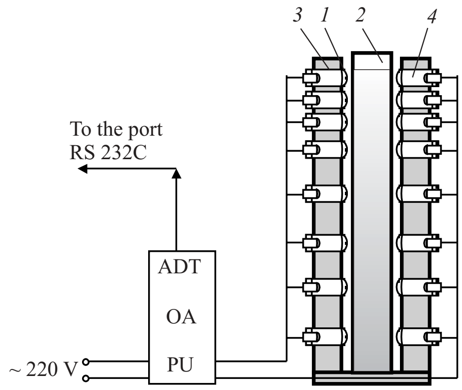

Fig. 1. Scheme of scanner sedimentograph SF-2.

ous analytical functions and determination of the distribution parameters;

– a block for matching different experiments: it is possible to simultaneously view, process, and compare the results of 2 to 10 experiments;

– a block for converting the measurement results into Excel tables and Word format;

– a reference database for disperse liquids with automated calculation of viscosity and density of liquids depending on temperature; a user manual for performing analysis.

One can work in various computer applications during analysis. The analysis results are automatically recorded in a file, and the user is informed of the completion of this operation by a sound signal. The duration of analysis is 5 – 10 min depending on the particle size.

The photosedimentation analysis is based on two physical processes:

– stationary sedimentation of particles in a viscous immobile medium according to the Stokes law;

– weakening of the light flux (extinction) as it is transmitted through the powder suspension layer according to the Bouguer – Lambert – Beer law.

The process of analysis using the photosedimentograph is as follows. A sample of the powder is thoroughly stirred in a dispersion liquid along the cuvette height. Such a liquid is usually distilled water or ethyl alcohol. Next, the device is switched on and analysis starts. Solid particle of different sizes precipitate under the gravity effect in accordance with the Stokes law. The coarse fractions precipitate first and the smaller ones after them. Accordingly, the concentration of particles along the cuvette height is changed. The cuvette is scanned along its height during the analysis. Each sensor registers extinction of the light flux with time. The digitized measurement data are put into the successive port. As a consequence of analysis, eight photosedimentograms are recorded and represent the variation of the photoflux depending on time. After the end of analysis the signals are mathematically processed and the granulometric composition is calculated.

Let us consider some processing algorithms and experimental results of analysis of ceramic materials.

The signal is smoothed at the first stage, which makes it possible to eliminate random fluctuations and decrease error generated by noise. Double smoothing is implemented: using a first degree polynomial based on five equidistant points and the first stage and exponential smoothing at the second stage to exclude accidental overshoots. Then the processed photosedimentogram is used to calculate the granulometric composition for a prescribed series of particles sizes. Original mathematical software has been developed that makes it possible to automatically determine the integral size distribution $R(x)$ of particles.

The problem can be formulated as follows. Given is a set of experimental points $R^{\mathrm{e}}(x_i)$ obtained in the course of analysis:

$$R_1^{\mathrm{e}}(x_1),\ R_2^{\mathrm{e}}(x_2),\ R_3^{\mathrm{e}}(x_3),\ \ldots,\ R_n^{\mathrm{e}}(x_n).$$

It is necessary to select a function of a prescribed type that gives the best approximation to the experimental data, i.e., to satisfy the condition

$$\sum_{i=1}^{n}\left\| R_i^{\mathrm{e}}(x_i) - R_i(x_i)\right\| \Rightarrow \min.$$

To solve this problem, it is necessary to set the method for measuring the distance from the experimental values to the approximated function, i.e., to set the norm. The successful solution of the problem to a large extent depends on the class (type) of the function and the type of the norm. The following types of norms are the most common: the sum of absolute values, the Euclidean norm, and the Chebyshev (uniform) norm.

The most common is the Euclidean norm. The least squares method is based on this norm. To show the effect of the norm choice on the parameters of approximation of the granulometric composition, the following calculation for two-parameter Plitt approximation was performed:

$$R(x) = \frac{100}{1 + (x / x_{50})^P},$$

where $R(x)$ is the integral distribution (full residues of particles sized from 0 to x), %; x is the current particle diameter, µm; x_{50} is the median size of particles, µm; P is the dimensionless parameter characterizing the spread with respect to the median size.

Table 1 shows the approximation parameters for various norms. The Chebyshev and the Euclidean norms yield close results, whereas the sum of absolute values provides for the most uniform approximation.

Thus solving the approximation problems using various norms makes it possible to obtain an analytic expression for the integral distribution of particle sizes.

TABLE 1

Particle size, μm	Integral distribution of particles, %			
	experimental data	Euclidean norm	sum of absolute values	Chebyshev norm
100	0.0	0.1	0.2	0.1
80	2.8	0.3	0.7	0.4
60	8.4	3.1	4.6	3.6
50	14.3	11.8	14.3	13.0
40	40.1	43.6	43.0	44.9
30	92.5	88.1	84.1	88.0
20	98.8	99.4	98.8	99.4
15	99.0	99.9	99.8	99.9
10	99.0	100.0	100.0	100.0
5	99.8	100.0	100.0	100.0
0	100.0	100.0	100.0	100.0

The following four types of two-parameter functions were used to describe the integral distributions: Plitt approximation, modified Rozin – Ramler approximation, normal-logarithmic law, and the Lynch function. The reason for taking four functions for approximation and not just one was due to the fact that apart from the median and the dispersion, the real distribution may have different asymmetry as well. These functions have different asymmetry values and satisfy the boundary conditions

$$\begin{cases} R(0) = 100; \\ R(x_{50}) = 50; \\ R(\infty) = 0. \end{cases}$$

In our case the experimental data can be best described using the Plitt approximation. For this function the sum of deviation squares is minimal. The division boundary x_{50} is close in all approximations. The approximation plots constructed based on the four specified functions agree well with the experimental values.

Let us consider the results of experimental analysis of grinding corundum micropowders produced by the Boksitogorskii Glinozem JSC. The granulometric compositions of

TABLE 2

Powder grade	Particle size, μm		
	d_3, not more than	d_{94}, at least	d_{50}
F230	82	34	53.0 (± 3.0)*
F240	70	28	44.5 (± 2.0)
F280	59	22	36.5 (± 1.5)
F320	49	16.5	29.2 (± 1.5)
F360	40	12	22.8 (± 1.5)
F400	32	8	17.3 (± 1.0)
F500	25	5	12.8 (± 1.0)
F600	19	3	9.3 (± 1.0)
F800	14	2	6.5 (± 1.0)
F1000	10	1	4.5 (± 0.8)

* Admissible deviation, μm.

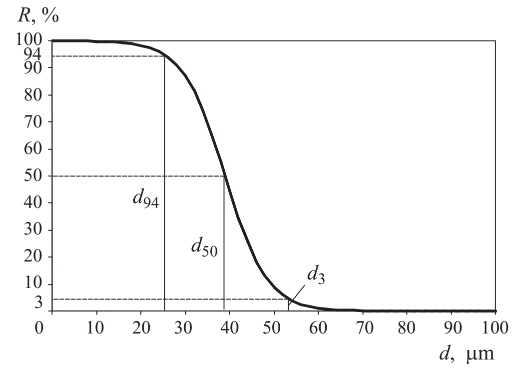

Fig. 2. Integral size distribution of particles $R(d)$.

the following grades of corundum powders were experimentally determined: F230, F240, F280, F320, F360, F400, F500, and F600. These powders are manufactured for export and ought to comply with the German standard (FEPE-Standard 42-D-1984, Teil 3-R1993), whose specific feature is that the size distribution of particles is characterized by three integral distribution parameters: d_{50}, d_3, and d_{94} (the diameters of particles, which content amounts to 50, 3, and 94%, respectively). The first parameters characterizes the median size and the two others indicate the spread with respect to the particular size. Figure 2 shows an example of integral distribution of particles by sizes and indicates the parameters that were determined, whereas Table 2 specifies the requirements imposed on various powder grades in accordance with the German standard.

It can be seen that each power grade ought to have not only a certain mean size but also a concentrated granular structure. Furthermore, the characteristic sizes of particles ought to be measured with a high degree of accuracy. The admissible deviation is within the limits of 3.0 to 0.8 μm.

Based on the performed studies, recommendations for selecting the optimum parameters for the Boksitogorskii Glinozem JSC were issued. The parameters recommended for analysis of grinding powders are listed in Table 3 and admissible errors in determining the parameters of the granulometric composition of powders based on the German standard FEPA and the actual errors of the device SF-2 are indicated in Table 4.

TABLE 3

Powder grade	Sample, g	Experiment duration, sec	Error magnitude (±), μm, based on parameters		
			Δd_{50}	Δd_3	Δd_{94}
F230	2.0	300	0.4	1.3	0.4
F280	2.0	300	0.2	0.3	0.2
F320	0.5	600	0.1	0.1	0.1
F360	0.2	900	0.1	0.2	0.1
F400	0.1	900	0.3	0.3	0.3
F500	0.1	1200	< 0.1	0.4	0.2
F600	0.1	1800	0.1	0.1	0.1

TABLE 4

Powder grade	Error magnitude (±), based on parameter Δd_{50}, μm	
	actual on instrument SF-2	admissible by standard FEPA
F230	0.4	3.0
F280	0.2	1.5
F320	0.1	1.5
F360	0.1	1.5
F400	0.3	1.0
F500	< 0.1	1.0
F600	0.1	1.0
F800	< 0.1	1.0

It follows from the data in Table 4 that the actual errors in determining the median size using the instrument SF-2 are significantly smaller than what is allowed by the German standard. This makes it possible to control the granulometric composition of grinding corundum micropowders at the Boksitogorskii Glinozem JSC.

The described instruments are used at nine factories in the ceramic, refractory, and other sectors of industry, including the DINUR JSC (Pervouralsk) and Nikom Refractories (Nizhny Tagil) for analysis of various refractories and slips, at the Chelyabinsk Electrode factory for analysis of graphite, at the Gora Khrustal'naya JSC (Ekaterinburg) for analysis of quartz sand, at the Boksitogorskii Glinozem JSC for quality control of export product, namely, corundum grinding powder to comply with the German standard FEPA, and at the departments of the Ural State Technical University and Perm State Technical University.

REFERENCES

1. P. A. Kouzov, *Principles of Analysis of Disperse Composition of Industrial Dust and Crushed Materials* [in Russian], Khimiya, Leningrad (1987).